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TEXTILE RESEARCH

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and for the Released Research Reports of
The Textile Foundation, Inc.

F. G. Cobb to Direct Research on Spun Rayon Warp Sizing

THE tentative appointment of F. Gordon Cobb as director of the research on the warp sizing of spun rayon and blends of cotton-spun rayon, to be conducted at North Carolina State College Textile School under the auspices of the Institute, is announced by Carl R. Harris, chairman of the Administrative Committee for the study. The appointment is "tentative" pending completion of the financing of this co-operative research, but as the fund is nearly subscribed the Committee is proceeding with plans to start active work.

An Open Research Conference

First, however, and as soon as the Administrative Committee has completed the compilation of a questionnaire on the subject of the study, to be submitted to subscribers for answer, the Committee will call an open conference of subscribers and others interested in this study to be held at N. C. State College Textile School. Heretofore conferences of subscribers after a co-operative research has been organized have always been confined to the subscribers. An exception is made in this case because the original conference, at which the Institute was asked to undertake the organization and financing of the research, was held in New York City and attended by comparatively few southern mill men, and also because this is the Institute's first research to be conducted in the South. At the coming conference non-subscribers will have the same opportunity as subscribers to hear the reports on plans for the study, to take part in the discussions relating to the final program, to visit the laboratory, and to learn at first hand of the research objectives and methods.

Mr. Cobb's Career

Mr. Cobb started his notable career in cotton manufacturing as an apprentice erector of Draper automatic looms in the South; was an overseer of weaving at 19, then successively superintendent of F. W. Poe Mfg. Co., Greenville, S. C., Inman (S. C.) Mills, and in 1919 the Springs Cotton Mills, Lancaster, S. C. He was with the latter for 16 years, advancing to general manager, vice-president and director, then retiring from active mill work excepting for some 18 months spent as vice-president in revamping Pomona Mills, Inc., Greensboro, N. C. Since then he has kept in touch with the industry as Market Counselor and textile consultant with *Cotton*, Atlanta, Ga.

He is a life member of the Southern Textile Association and has served this body as president and executive secretary, also receiving its S. B. Alexander gold medal in 1926 "for greatest service to the southern textile industry" in that year.

Mr. Cobb brings to his new duties a broad knowledge of the subject to be investigated, the executive ability to direct the work toward its objectives without a waste of time and effort on fundamentals, and has associated with him in an advisory capacity a group of scientists and manufacturers. As a manufacturer he was noted for his knowledge of warp sizing materials and methods. When most mills were making their own size mixtures he collaborated with two firms in perfecting sizing formulas that are still widely used. When the late Dr. Catheart was engaged in developing thin-boiling starches the practical tests were conducted by Mr. Cobb.

* * *

Open Meeting of Committee D-13 at Charlotte, N. C.

COMMITTEE D–13 (Textiles) of the American Society for Testing Materials is to hold its Spring Meeting on March 13 and 14 at Hotel Charlotte, Charlotte, N. C. It will be the first meeting to be held by the Committee in the South, and will be open to all who are interested in its work of developing and promulgating methods of test and textile standards. The Committee believes that it can better serve the purposes for which it was founded in 1914 by broadening its scope geographically, although at present it has many southern members.

While the usual sub-committee meetings will be held on both days there will be an afternoon session each day for the presentation of technical papers and on the evening of March 13 there will be an informal banquet. Those interested in attending and desiring program should address W. H. Whitcomb, Secretary, 41 Norman Ave., Cranston, R. I.

* * *

Co-operative Research for Textile Profits

CO–OPERATIVE RESEARCH, on which other industries than textiles are spending approximately $200,000,000 a year, is advocated by Fuller E. Callaway, Jr., president, Callaway Mills, Inc., La Grange, Ga., in an article in the recent "Textile Profits Issue" of the *Daily News Record*, "as a desirable, possibly a necessary and preservative course for the cotton textile industry."

After an interesting and effective summary of old industries revivified by research and the many new industries created by research he concludes the article as follows:

"Present day competition is not only between companies but also between industries. Reinforced concrete competes with structural steel; fuel oil with coal; welding with riveting; jute, paper, plastics with cotton. As a result many industries have found it desirable or necessary to combine their resources for the common good.

(*Continued on Page 192*)

Combination of Wool Protein with Acid and Base: Hydrochloric Acid and Potassium Hydroxide*

By JACINTO STEINHARDT and MILTON HARRIS †

A Textile Foundation Publication

Abstract

THE protein material of which wool is principally composed possesses a variety of chemical groups capable of combining with acids and bases. The state of combination of these groups is intimately related to the ability of wool to absorb moisture and dyes, its elastic and tensile properties, and its behavior in carbonizing, scouring and milling. In order to form a background for investigations relating to these technologically significant properties of wool, an extensive investigation of the acidic and basic properties of the fibres has been undertaken. Information obtained from such an investigation should also aid considerably in the further elucidation of the chemical and physical structure of wool fibres.

The present study is concerned with the description of the relation between amounts of hydrochloric acid and potassium hydroxide bound by wool and the concentration of these substances in solution. The effect of the presence of an added neutral salt, potassium chloride, on this relation has also been studied. Al-

* Part of the material in the present paper was presented at the meeting of the American Society of Biological Chemists at Toronto, Canada, in April, 1939.

† Research Associates at the National Bureau of Standards representing the Textile Foundation.

though the maximum amounts of acid or base bound are independent of the presence or absence of salt, the way in which this maximum is approached depends very greatly on the presence of salt, and varies with the amount. Thus, the range of concentrations of acid in which the amount of acid bound increases from zero to near its maximum value is relatively narrow when salt is absent. Since this is also true of combination with base there is a wide region in the middle of the acid-base scale in which no combination with either can be detected. In the presence of even small concentrations of salt, the course of combination is more gradual, and the point at which no combination with either acid or base occurs is sharply defined. The higher the concentration of salt present, the lower is the concentration of acid or base at which any given amount of either is combined. This dependence is so marked that, in dilute solutions, doubling or tripling the concentration of salt has practically the same effect as doubling or tripling the concentration of acid or base. It is shown that all these effects of salt are manifestations of the same restriction: the fibre must combine with or adsorb equivalent quantities of both positive and negative ions. Thus, its ability to combine with hydrogen ions is limited by the simultaneous availability of chloride ions. By interpreting the combination with these accessory ions in terms of the partial dissociation of protein salts formed by combination with acid or base, the details of the difference between the curves of combination in the absence of added salt and in the presence of different concentrations of added salt are fully explained. This interpretation also leads to the prediction that the data of acid combination obtained in the presence of high concentrations of salt

should correspond very closely with the results that would be obtained if wool could be studied in the dissolved state. By comparing the data obtained for wool with data for a soluble protein, egg albumin, which contains the same proportion of different acidic groups, this prediction is verified. On the basis of this comparison and by analogy with similar work on other soluble proteins, the curves relating the amount of acid combined to degree of acidity are resolved into component parts which are consistent with previously published figures for the amino-acid composition of the fibre.

The demonstration that the combination of anions with the fibre is a limiting factor in the combination with acid may prove to be significant in the process of acid dyeing. In this process, competition between the colored dye anions and the simple anions of the commonly used Glauber's salt may play a part.

I. Introduction

THE nature of the dependence on pH of the degree of combination of acid and base by such insoluble proteins as wool, silk, and collagen, differs in certain characteristic ways from that of soluble proteins. Examination of measurements reported by Speakman and Hirst,[42] Lloyd and Bidder,[34] and others leads to the conclusion that the titration curves of these three proteins, determined in the absence of salt, are distinguished from those of all soluble proteins so far studied * under the same conditions in three principal respects:

(1) They show a long region of little or no acid- or base-binding capacity, more or less symmetrically placed about the point of neutrality. This is true even when, as in the case of wool and silk, they contain appreciable quantities of histidine which, because of its imidazole group, should function as a buffer in this region.

(2) The amounts of acid and base bound increase sharply as the extremes of the pH scale are approached and reach their respective maximum values at about the same pH as in the case of soluble proteins. Thus, the titration curves are steeper than those of soluble proteins, or of soluble polybasic acids in general.

(3) The pH values at which half of the maximum amount of acid or base is combined (a convenient measure of the position of the curve and, in

* Except those, such as hemoglobin, in which irreversible effects occur.[10, 37]

simple substances, directly related to the acid strengths of the groups titrated), are shifted considerably toward the two extremes of the pH scale. Thus, the S-shaped portions of the curves found on the acid side of pH 7 are centered, in the case of almost all dissolved proteins, about a pH very close to 4.0, but in earlier published work on wool this point appears to be 2.3.[43, 44, 45] If these curves are regarded as resulting from the titration of many groups which possess identical dissociation constants this difference in the position of the midpoints would represent a fifty-fold increase in the acid strength of the insoluble protein, although its dissociating groups are presumed to be the same as those present in proteins which may be titrated in the dissolved state.

This description of the striking differences between the titration curves of soluble and insoluble proteins is made more concrete by the comparison in Fig. 1 of the data for wool (obtained as described in the experimental

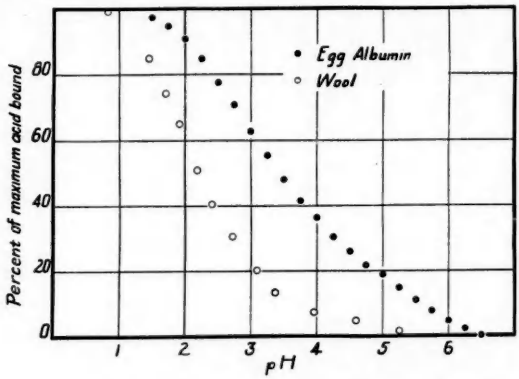

Fig. 1. Comparison of the curves of acid combination of wool and of egg albumin at 25°, in the absence of added salt.

section) with similar data of Kekwick and Cannan[31] for the soluble protein, egg albumin, both at 25° C. The latter are chosen because of their general excellence, and the close similarity in relative composition of egg albumin and wool, with respect to those amino acids (aspartic acid, glutamic acid and histidine) which should affect the shape and position of their titration curves * in the acid region, although these proteins differ considerably in their combining weights. The same scale of ordinates is used for the two curves by plotting, instead of the amount of acid combined per gram by each protein at each pH, the fractions of the total amounts of acid which are required to bring them from a condition of equilibrium with a solution at pH 6.4 to one at pH 1 (the reason for choosing pH 6.4 will be apparent later). These curves are typical of those obtained over this range of pH

* The position and form of the acid titration curves are determined by the carboxyl groups of the protein rather than by the amino groups, because the latter are present as internally ionized ammonium-like salts. Thus the process of acid combination consists in replacing a proton on an ionized carboxyl group. The maximum amount of acid combined is, of course, determined by the number of amino groups.[3, 7, 24]

with proteins of the two classes, and illustrate: (1) the long region, starting below pH 5 (and extending to pH 10) in which practically no acid combines with wool; (2) the much greater steepness of the curve for wool; (3) the much greater acidity at which half the maximum amount of acid is combined.

Cannan has shown that this portion of the curve for *egg albumin* (after correction for the small amount of acid combination due to histidine) is readily described in terms of the law of mass action, using only a single dissociation constant, if allowance is made, in a semi-empirical way, for the effect of electrostatic interaction between the ionized carboxyl groups on their respective dissociations.[4] Clearly, if a similar analysis is to be applied to the data for wool, additional factors must be taken into account. A possible clue to the nature of the difference is suggested by the fact that the steeper slope characterizing the curves for the insoluble proteins may be described to a good degree of approximation by substituting $(H^+)^2$ for (H^+) in the mass law expression for egg albumin used by Cannan.

It seems likely that these differences, characteristic not only of wool but of other high-molecular insoluble proteins as well,[24] are necessarily associated with the presence of a separate phase. Nevertheless, many proteins which are relatively insoluble at their isoelectric points have hitherto been titrated in the presence of much undissolved material over at least part of the total range of the titration. Singularities and discontinuities, which may not be an essential part of the titration phenomena but rather the result of the disappearance or appearance of a second phase, have appeared in these data.[5]

Among those who have recognized the existence of these differences between the titration curves of dissolved and undissolved proteins, Speakman and his collaborators[42, 44, 45] and Lloyd and Bidder,[24] have preferred to attribute them to fundamental differences between the structures of insoluble and soluble proteins rather than to factors introduced by the existence of two-phase equilibria. Thus Speakman appears to believe that salt linkages, between acidic and basic groups, prevent the functioning of these groups in their customary ranges of acidity because certain minimal acid or base concentrations must be exceeded before such salt linkages are broken. This is equivalent to postulating a large charge effect, transmitted through the solvent medium, on the dissociations of these groups. In view of the high dielectric constant of water, the postulated magnitude of this effect is open to question.[2] In addition, Lloyd and Bidder, working with collagen, gelatin, silk, and horse-hair, attribute a part of the inactivity of acidic and basic groups at reactions near neutrality to the existence of vaguely-defined covalent linkages between adjacent polypeptide chains, which render the groups in question unavailable until these linkages are destroyed by exposure to sufficiently concentrated acid or alkali.

Nevertheless all of these authors, and others who have been concerned with the electrochemical and osmotic behavior of insoluble proteins, have recognized that account must be taken of the presence of two phases, and have had recourse to the usual method of describing such phase equilibria by means of the Donnan equations,[12, 13] at least in a qualitative way. A *quantitative* analysis of the titration curve, sufficient to decide the extent to which the existence of two phases eliminates the need to consider other factors of the kind mentioned above, has not hitherto been made. The reason for this is not far to seek: there are grave difficulties in defining, without

arbitrariness, the compositions of the phases in applying the equations of the Donnan equilibrium to the cases in question. In the present paper new data on the combination of wool with both acid and base are described in terms of a more readily defined and simpler concept. This treatment does not pretend to supplant the treatment of Donnan in other cases of two-phase equilibrium for which exact information as to the composition of clearly delimited phases makes possible its proper application.

As a result of certain theoretical considerations which are discussed in Section IV, it appeared likely that data obtained in the presence of neutral salt at constant ionic strength might shed light on the cause of the differences apparent between curves obtained with acid alone (Fig. 1). The measurements to be described here, most of which were made at 0° C. to avoid complications introduced by hydrolytic decomposition, have led to a simple and general way of relating these differences to the conditions under which insoluble proteins such as fibres must be titrated. It seems probable that the resulting viewpoint has consequences for many phenomena characteristic of soluble as well as of insoluble proteins. Thus, for example, in the study of the effect of pH upon protein solubility a condition necessary for measuring the phenomenon is the presence of the solid phase.

II. Experimental Procedure

1. Material

Raw wool fibres were cut into root and tip portions, and the latter, which are contaminated and partially decomposed, were discarded. The root portion was purified by washing in cold ether, extracting for 16 hours, first with alcohol and then with ether, at temperatures below 35° C., and finally washing repeatedly in distilled water. It was carded by hand or on a sample carding machine,* rewashed in distilled water, air-dried, and finally conditioned at 21° C. and 65% relative humidity before weighing. Analyses for total nitrogen, amide nitrogen, primary amino nitrogen, sulfur, and cystine gave nearly identical results with all the batches of wool used. Analyses for ash were made by combustion, and by an adaptation of the electrodialysis method of Joseph and Stadie.[30, 48] Most of the data on combination of acid at 0° were obtained with wool containing only 0.10% ash. No effort was made to remove this ash and no correction was applied for its effect (shown to be very small, see III–I) on the amount of acid combined. The remaining data, obtained with material of higher ash content (0.26%), were corrected for the alkali equivalence of the ash by subtracting from or adding to the measured acid- or base-binding capacity an amount determined by direct comparison with the low-ash wool, occasionally supplemented by other methods of estimation.† The moisture content of the fibres used in each experiment was determined by drying representative samples for two hours in a vacuum oven at 105° C. The data are expressed as acid or base bound per gram of dry wool.

2. Methods

(*a*) *Titration with acid.*—Samples of wool weighing either 1.2 or 2.0 g and containing from 13.1 to 15.0% of moisture, were placed in 100-ml por-

* Thanks are due to the Eavenson & Levering Company of Camden, N. J., for carding four batches of wool used in this work.

† The principal alternative method used was an electrodialytic estimation of combined base, by an adaptation of the method of Joseph and Stadie.[30]

tions of acid solutions at the temperature of the experiment (0° or 25° C). The solutions contained varying amounts of hydrochloric acid and sufficient potassium chloride to bring them, after absorption of acid by the fibres, to predetermined ionic strengths between 0.005 and 1.0. Sets of solutions containing only HCl, without added salt, were also used. Rapid and complete wetting-out of the wool was facilitated by pumping off air with a filter pump. Repeated preliminary sampling showed that during immersion over a period of 48 hours at the lower temperature, there was a perceptible drift of pH in solutions from which a large fraction of the total acid was removed by combining with the wool. Therefore, in order to assure attainment of equilibrium, the fibres were exposed to the solutions at either temperature for periods of from 48 to 75 hours before samples were withdrawn. Portions of each solution, transferred to stoppered Pyrex test-tubes, were allowed to come to room temperature (22° C–31° C) and the pH of each was determined. Aliquots of the original solution and of the solution in equilibrium with the wool were titrated with sodium hydroxide, using bromcresol purple as indicator. From the difference between these titers, the weight of the sample, and its content of moisture, the amount of acid taken up per gram of dry wool was calculated. Since differences in titers are involved, and since these differences do not change in direct proportion to the total concentration of acid, the measurements made in the most acid solutions are most subject to error. This limitation of accuracy was partly offset by using the larger wool samples in the most acid solutions.

The chloride disappearing from solution was determined by titrating the same aliquots, after neutralization, with 0.1 *M* silver nitrate. Sufficient potassium chromate solution to bring the chromate concentration above .01 *M* was used as indicator. Errors caused by end-point uncertainties were reduced by matching the colors of the aliquots with one another.

In calculating the hydrogen ion and chloride ion bound by the fibres from the difference in titers of the solutions, proper account was taken of the effect of water absorption by the fibres on the final concentration of acid. With the ratios of fibres to solutions used, this correction was negligible except when the acid concentration exceeded 0.1 *M*, but the importance of this procedure has been amply demonstrated by critical experiments with ratios more favorable to the detection of effects due to moisture absorption. In calculating these small corrections, it has been assumed that the hydration of the fibre is the same in acid solutions as it is in water, and that no hydrochloric acid is dissolved in the water taken up. Recent unpublished measurements on cotton fibres in this laboratory indicate that the latter assumption may not be strictly true, but only a slight over-correction results from its use. Omission of the correction would result in an apparent decrease in the acid bound when the concentrations of acid exceeded 0.2 *M*.

In order to determine whether prolonged exposure to acid produced significant hydrolysis of amide bonds, other aliquots from each solution were tested for ammonia with the Nessler reagent of Koch and McMeekin.[53] At 0° C, only the highest acidities used (over 0.2 *M*) produced a detectable amount of ammonia (.06 mg. per g.). The effect of this amount on the titration curve is practically negligible. At 25° C, concentrations over 0.1 *M* produced sufficient ammonia to account for 0.01–0.03 millimole of acid combined. This is equivalent to hydrolyzing from 1 to 2% of the amide groups present. Tests with the phenol reagent of Folin and Ciocalteu[14] showed the presence of traces of a reducing substance in the

most strongly acid solutions. This appeared to be present in the wool before treatment with acid rather than to result from decomposition since the maximum amount found was the same at both temperatures. Negative sulfide and biuret tests on the solutions showed that no appreciable dissolution of decomposition had occurred.

(*b*) *Titration with base.*—Portions of wool weighing either 3.0 or 4.0 g were placed in carbonate-free alkaline solutions (usually in volumes of 200 ml) at 0° C. The solutions contained varying amounts of potassium hydroxide and sufficient potassium chloride to bring the total ionic strengths to predetermined values of 0.02 or 0.2 *M*. Sets of solutions without added salt were also used. The larger samples were used in the most alkaline solutions. Although it is very desirable to limit the time of exposure to alkali to a minimum in order to minimize the amount of decomposition, control experiments at acidities at which decomposition was negligibly slow showed that periods of immersion of at least 18 hours were required to attain equilibrium. Approximately 20 hours was allowed in all the experiments reported, with the exception of those mentioned later.

In determining the amounts of base bound the procedure of Harris and Rutherford [25] was followed except for the substitution of bromcresol purple for methyl red as indicator, and the use of nesslerization for the determination of dissolved protein. Additional aliquots were used for immediate measurement of pH at the temperature of the experiment. The use of bromcresol purple reduced the error introduced on the acid side of pH 6 by the buffering capacity of dissolved protein in those solutions in which appreciable decomposition has occurred.*

A slight modification of the method of calculating the corrections was introduced. Comparisons of the ratio of dissolved nitrogen, after Kjeldahl digestion, to dissolved inorganic sulfur with the same ratio in the intact protein indicated that a small fraction (usually from one-tenth to one-fifth) of the inorganic sulfur in each solution was derived from the wool that had dissolved. Therefore, only the remaining eight- or nine-tenths of the inorganic sulfur in solution was produced by hydrolysis of disulfide bonds *in the undissolved wool.* Only the latter portion should be included in the correction for disulfide bonds broken since the base taken up by the dissolved protein, including that taken up by the sulfhydryl groups formed by hydrolysis of its own cystine, is not measured by the differential titration method used in this work.

This modified correction was shown to be justified by control measurements with several different concentrations of potassium hydroxide between 0.05 and 0.30 *M*, and with periods of immersion between 1.5 and 27 hours at 0° C. These measurements showed that with the highest concentrations there is a measurable increase in the amounts of base combined up to at least ten hours, and that after this period, the amount bound does not change if the constantly increasing corrections for decomposition are properly made. If on the other hand the corrections are applied as in the method of Harris and Rutherford, the calculated amount of base bound decreases somewhat on prolonged exposure to strongly alkaline solutions. This would be the expected result of over-correction when the amounts of decomposition become large. The same effect is evident when results at different concentrations

* Buffering capacity on the alkaline side of the pH for zero combination is taken care of by the methods of correction for the effects of decomposition used by these authors.

of potassium hydroxide, obtained after the same times of exposure, are compared. The calculated amounts of base bound tend to *decrease* at the highest concentrations if the correction for *all* the inorganic sulfur is applied.

At 0° C the largest correction, 0.28 milliequivalent per gram, corresponded to the destruction of approximately one-half of the disulfide bonds and the dissolving of $\frac{1}{12}$ of the total protein. In similar controls carried out at 25° C as much as 85% of the cystine was destroyed, and up to one-sixth of the protein dissolved. With these larger amounts of decomposition, the necessity for modification of the method of Harris and Rutherford was even clearer. Thus, while the modification described here gave consistent results at the two temperatures, the unmodified corrections gave lower values at high concentrations of potassium hydroxide at the higher temperature.

Since the earlier measurements of Harris and Rutherford were obtained, for the most part, after exposure of wool to alkali for two hours, and since the corrections subtracted were slightly high, their values for the amounts of base bound are probably a little lower than the true equilibrium values. The data of the present investigation indicate that the maximum possible amount of base has not been bound even at potassium hydroxide concentrations of 0.3 *M*. However, the protein may be modified by decomposition of bonds other than disulfide when these or higher concentrations of alkali are used, and the methods of correction applied here may no longer suffice.

In view of the magnitude of the corrections described, no additional corrections for the effect of water absorption by the fibres on the calculated amount of combined base were applied. The small effect of this correction is within the error of the values given.

(*c*) *Measurement of pH.*—Measurements of pH were made with the glass electrode assembly developed by MacInnes and Belcher,[35] connected with a thermionic potentiometer which employed a cathode-ray "eye" as null indicator. The potentiometer, arranged to read directly in pH at temperatures between 0° and 30° C, could be set reproducibly to 0.002 pH unit when measuring buffered solutions. The following solutions[29] were employed in establishing the pH scale in terms of which the measurements at 25° are expressed: (a) 0.1 *M* hydrochloric acid, and (b) 0.01 *M* hydrochloric acid plus 0.09 *M* potassium chloride, using the values 1.085 and 2.078, (c) 0.05 *M* potassium hydrogen phthalate, using the value 4.008, (d) 0.01 *M* acetic acid plus 0.1 *M* sodium acetate, using the value 4.648, (e) 0.05 *M* sodium tetraborate, with the pH value 9.180. In addition, 0.05 *M* potassium hydroxide was used. A pH of 12.61 was assigned to this solution, consistent with the activity coefficient (0.824) determined by Harned and Cook[29] and the value of Harned and Hamer[22] for the ion product of water (pKw = 13.997).

Calibration at 0° C was carried out with the same solutions, omitting the sodium tetraborate. The same pH values were assigned to the hydrochloric acid and phthalate[18] solutions at both temperatures, but the value assigned to the acetate buffer at 0° C (4.673) was estimated by adding to the value at 25° C the logarithm of the ratio of the dissociation constants at the two temperatures. The negligible effect of temperature on the activity coefficients of the ions in these solutions is thus not taken into account in the values assigned. The pH assigned to 0.05 *M* potassium hydroxide at 0° C was 13.56, estimated from the value of Harned and Hamer at this temperature for pKw (14.94) and the activity coefficient (0.829) determined by Harned and Cook.

When the pH-meter was set at the proper pH with any of these standards except the most alkaline one, each of the other four buffers could be read to within 0.01 pH unit of the values assigned to them. Readings in alkaline solutions required adjustment for the departure, at pH greater than 9, of the slope of the pH-e.m.f. function from its theoretical value. This adjustment was carried out as follows: after setting the pH-meter to read 4.008 with the phthalate buffer in the electrode vessel, the pH reading of 0.05 *M* potassium hydroxide was determined, and subtracted from the calculated correct reading. The difference (never over 0.26 pH unit at 0° C) was assumed to be uniformly cumulative over the pH interval of 8.55 units. This is equivalent to assuming that over this range of pH the proportionality between e.m.f. and pH is lower than the proper value of RT/F by about 3%. This assumption leads to adjustments which are subject to most error at pH 7 to pH 10. In this interval only a few readings were taken; and owing to the relatively small slope of this part of the titration curve, a small error in pH has very little effect on its shape or position. The pH values reported for alkaline solutions are less certain than those in the acid range because of these adjustments (based in part on a *calculated* pH) and because of some hysteresis of the glass membrane after exposure to concentrated alkali. The latter sometimes introduced a discrepancy of .03 to .04 pH unit, apparent when the measurements of acid standards were repeated after an alkaline series. In agreement with the work of Dole and Wiener [11] the required adjustments and the residual uncertainties are larger at the higher temperature.

The pH values of alkaline solutions were measured with the electrode assembly mounted in an air thermostat at either 3° C or 25° C. Solutions were introduced and manipulations made from the outside. The results given at 0° C are calculated from measurements made at the lower temperature. Practically all acid solutions were measured at room temperature. Since many of the solutions were very poorly buffered, the accuracy of a large number of the measured pH values was tested by direct comparison with the calculated values based on the titer of the solutions and the activity coefficients of the acid or base.[19, 20, 21, 22, 26] With solutions more acid than pH 3.5 at ionic strengths below 0.1 *M*, agreement to .01 pH unit was usually found. The presence of *traces* of a soluble buffering substance derived from the wool reduced this agreement in the range pH 4 to 10, but in the absence of wool this agreement extended to pH 4.5, beyond which the concentration of HCl could not be accurately determined. The stability and reproducibility of the measurements below pH 6 render it very probable that the same high accuracy prevails up to this value, even in the presence of wool. Measurements between pH 6 and 9 required almost continuous flushing of the electrode vessel, and are less certain. pH values for solutions containing potassium hydroxide in concentrations of 0.1 *M* or more were calculated from their titers, allowance being made for the contribution to the titer of any hydrosulfide present. Acid solutions with an ionic strength of more than 0.1 required application of a small empirical correction (between 0.02 pH at ionic strength 0.2 and 0.11 pH at ionic strength 1.0) due to the difference in ionic strength between the standardizing buffers and the solutions measured.

(*d*) *Control of temperature.*—A well-stirred 200-liter water-thermostat equipped with cooling coils, 300-watt heating element, mercury thermoregulator, and time-delay relay, kept the temperature constant at 25.00° ± .02° C. No thermoregulator was used at 0° C; ice was allowed to form on the coils

as the result of intermittent operation of the cooling unit. With a large amount of ice present, the temperature remained constant to ± .01° C.

References

1. Adams, E. Q. *J. Am. Chem. Soc., 38*, 1503, 1916.
2. Bernal, J. D. Lecture at Royal Inst., London, Jan. 27, 1939. Summarized in *J. Soc. Dyers Col., 55*, 308, 1939.
3. Bjerrum, N. *Z. physik. Chem., 104*, 147, 1923.
4. Cannan, R. K. Cold Spring Harbor Symposia in Quantitative Biology, *6*, 1, 1938.
5. Cohn, E. J. *Physiol. Rev., 5*, 349, 1925.
6. Cohn, E. J. *Ergebn. Physiol., 33*, 781, 1931.
7. Cohn, E. J. *Chem. Rev., 19*, 241, 1936.
8. Cohn, E. J. Cold Spring Harbor Symposia in Quantitative Biology, *6*, 8, 1938.
9. Cohn, E. J., Edsall, J. T., and Blanchard, M. H. *J. Biol. Chem., 105*, 319, 1934.
10. Cohn, E. J., Green, A. A., and Blanchard, M. H. *J. Am. Chem. Soc., 59*, 509, 1937.
11. Dole, M., and Wiener, B. Z. *Trans. Electrochem. Soc., 72*, 25, 1937.
12. Donnan, F. G. *Z. physik. Chem., 168A*, 369, 1934.
13. Donnan, F. G., and Guggenheim, E. A. *Z. physik. Chem., 162A*, 346, 1932.
14. Folin, O., and Ciocalteu, V. *J. Biol. Chem., 73*, 627, 1927.
15. Fruton, J. S., and Lavin, G. I. *J. Biol. Chem., 130*, 375, 1939.
16. Graff, S., Maculla, E., and Graff, A. M. *J. Biol. Chem., 121*, 71, 1937.
17. Green, A. A. *J. Am. Chem. Soc., 60*, 1108, 1938.
18. Hamer, W. J. Publication pending.
19. Harned, H. S. *J. Am. Chem. Soc., 47*, 689, 1925.
20. Harned, H. S., and Cook, M. A. *J. Am. Chem. Soc., 59*, 496, 1937.
21. Harned, H. S., and Ehlers, R. W. *J. Am. Chem. Soc., 55*, 2179, 1933.
22. Harned, H. S., and Hamer, W. J. *J. Am. Chem. Soc., 55*, 2194, 1933.
23. Hammersten, E. *Biochem. Z., 144*, 383, 1924.
24. Harris, L. J., and Birch, T. W. *Biochem. J., 24*, 1080, 1930.
25. Harris, M., and Rutherford, H. A. *N. B. S. J. Rsch., 22*, 535, 1939.
26. Hawkins, J. E. *J. Am. Chem. Soc., 54*, 4480, 1932.
27. Hitchcock, D. I. Cold Spring Harbor Symposia in Quantitative Biology, *6*, 24, 1938.
28. Hitchcock, D. I., in Schmidt, C. L. A. "Chemistry of the Amino-Acids and Proteins." Charles C. Thomas, Springfield, Illinois, 1937.
29. Hitchcock, D. I., and Taylor, A. C. *J. Am. Chem. Soc., 59*, 1812, 1937.
30. Joseph, N., and Stadie, W. C. *J. Biol. Chem., 125*, 795, 1938.
31. Kekwick, R. A., and Cannan, R. K. *Biochem. J., 30*, 227, 1936.
32. Kern, W. *Z. physik. Chem., 189A*, 249, 1938.
33. Koch, F. C., and McMeekin, T. L. *J. Am. Chem. Soc., 46*, 2066, 1925.
34. Lloyd, D. J., and Bidder, P. B. *Trans. Faraday Soc., 31*, 864, 1934.

35. MacInnes, D. A., and Belcher, D. P. *J. Am. Chem. Soc., 53,* 3315, 1931.

36. MacMahon, P. R., and Speakman, J. B. *Trans. Faraday Soc., 33,* 844, 1937.

37. Morrison, D. B., and Williams, E. F., Jr. Proc. Am. Soc. Biol. Chemists, *J. Biol. Chem., 123,* lxxxvii, 1938.

38. Rutherford, H. A., and Harris, M. In preparation for publication.

39. Rutherford, H. A., Harris, M., and Smith, A. L. *N. B. S. J. Rsch., 19,* 467, 1937.

40. Simms, H. S. *J. Am. Chem. Soc., 48,* 1239, 1926.

41. Simms, H. S., and Levene, P. A. *J. Biol. Chem., 70,* 319, 1926.

42. Sookne, A. M., Steinhardt, J., and Harris, M. In preparation for publication.

43. Speakman, J. B., and Hirst, M. C. *Trans. Faraday Soc., 29,* 148, 1933.

44. Speakman, J. B., and Stott, E. *Trans. Faraday Soc., 30,* 539, 1934.

45. Speakman, J. B., and Stott, E. *Trans. Faraday Soc., 31,* 1425, 1935.

46. Speakman, J. B., and Townend, F. *Nature, 139,* 411, 1937.

47. Steinhardt, J. *J. Biol. Chem., 129,* 135, 1939.

48. Steinhardt, J. In preparation for publication.

49. von Muralt, A. L. *J. Am. Chem. Soc., 52,* 3518, 1930.

50. Vickery, H. S., and Bloch, R. J. *J. Biol. Chem., 86,* 107, 1930.

51. Weber, H. H. *Biochem. Z., 189,* 381, 1927.

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"Among those which have spent the largest amounts on co-operative research are American Gas Assn., American Petroleum Institute, California Fruit Growers Exchange, National Canners' Assn., Portland Cement Assn., and Utilities Research Commission. There are many others.

"I regard such a co-operative endeavor as a desirable, possibly a necessary and preservative course for the cotton textile industry.

"The other industries of the United States are spending approximately 200 million dollars a year on research. If all the textile mills in the country would pay an amount equal to one per cent of their net invoices into a fund to promote research for the benefit of the industry, we would have a fund of approximately 25 million dollars a year.

"With this the textile industry would not only get on its feet, but would soon be riding the crest of the wave."

The Sampling of Domestic Wool for Shrinkage Determination*

By ROBERT H. BURNS †

Summary

The terms shrinkage, yield, and clean content are defined. The methods of obtaining shrinkages on lots of wool are explained. The research work completed with wool shrinkage tests in the past is reviewed.

The method of selecting the sub-lot and the samples from the sub-lot is described, and the methods of scouring at the laboratory and the mill are explained. The shrinkages of the small samples are compared with those of the sub-lot and the entire lot.

The grading and sorting of the sub-lots are compared with that of the entire lots. The grading of 10 bags, of five bags, and of every nth fleece is compared to that of an entire clip.

The grading of the first bag sheared from each band is found usually to be coarser than that of the last bag sheared from each band, and the cause is explained.

Figures are given for the percentage of grease in the scoured wool. The variations in shrinkage of samples drawn by different operators, and between the first and fourth samples drawn are given, and are found not to be significant.

Introduction

THE efficient production of wool requires a definite knowledge of the ranges of quality occurring in the clip before any improvement can be made through breeding and management. A knowledge of the product which he is selling is helpful to both the seller and the buyer. The wool grower has become increasingly interested in the inherent value and variation in value of the fleeces in his clip, because he must KNOW and RAISE a higher quality of wool in order to run his business efficiently and profitably.

Good merchandising of wool requires a knowledge of its grade, uniformity, and shrinkage. The wool grower is interested not only in the merchandising of his wool clip, but is also vitally concerned with the uni-

* This paper is a progress report on a research project directed by Dr. Burns while he was a member of the staff of the U. S. Department of Agriculture. The work is being carried on by the Agricultural Marketing Service of the Department in co-operation with wool-growing states and the textile industry.

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formity and quality of his clip. With such knowledge the grower is equipped to set up a definite goal of wool improvement through breeding and management which should result in a higher income from the wool produced by each sheep in his flock.

The information which the grower obtains from the buyer concerning his clip practically always is biased, and the only other source of information is for the grower to have the wool tested by commercial scouring plants or through his state Agricultural Experiment Station. The experiment stations and government agencies have made shrinkage tests of wools for a number of years, but very few results were available on the direct comparison of small samples and entire lots, or clips of wool. In so far as the writer is aware, from the literature, there is only one test reported, and that is the one given by Dantzer[1] (1922) who made a series of tests for the French Government in which small samples were compared with the entire lot. He tested four 500-gram (1.1 pound) samples drawn from a lot of wool weighing 48,044 pounds. The samples gave a shrinkage of 43.4% while the figure for the entire lot was 51.4%, a difference of around 8%. It is interesting to note that the experts' estimates of shrinkage on the same lot of wool were in error by around 6%. The method of taking the samples is not given except to mention that they were representative. The ratio of sample weight to lot weight was extremely high.

Although samples have been compared with each other and with individual and groups of fleeces from which they were taken, never, in so far as reported in the literature, have any samples or fleeces been compared with entire clips.

Because of this lack of information the small samples have been open to criticism as being unrepresentative, and it seemed highly desirable to make a controlled sampling experiment in which small samples taken in an orderly manner were compared with the entire lot when processed at the mill, which is generally accepted as the ultimate check of the yield of clean wool.

Such a program has been undertaken by the U. S. Department of Agriculture in co-operation with the wool growing states, and the present paper is a *progress report* on the first year's work on this program, carried on under the writer's direction while he was connected with the U. S. Department of Agriculture. It is not to be considered as a final report on this work, but the results are interesting and consistent and indicate that a method of taking small samples can be worked out which will give quite representative shrinkage results for a clip of wool.

Shrinkage Defined

The fleece of the sheep contains varying amounts of extraneous material, such as dirt, sand, burrs, seeds, greasewood, cactus and paint; and indigenous material, such as sweat, excreta and grease.

The commercial value of a fleece or a clip of wool is, to a large extent, controlled by the amount of clean fibre that it yields. The weight of clean wool fibre, as expressed by the percentage it makes up of the original weight

[1] Dantzer, M. Robert. 1922. "The Experience of France in Testing Wool for Shrinkage during the War." Hearings on the Tariff Act of 1921. Sen. Doc. 108, 67th Congress, 2d Session, Vol. V, pp. 3630–3634. Washington: Government Printing Office.

of greasy wool, is spoken of as the YIELD. When an allowance is made for the impurities remaining in the scoured wool after it is washed, the term CLEAN CONTENT is used to describe the clean wool which contains no impurities. The percentage of the original weight of the greasy wool removed by scouring is spoken of as the SHRINKAGE. The commercial shrinkage is based on the billed weight of grease wool. The commercial value of a clip or lot of wool cannot be ascertained until the shrinkage and grade are known or estimated by competent wool handlers.

Experimental Procedure

Original bag and graded lots of wool were sampled at the mill prior to processing in the following manner:

The first step was to take out of the lot approximately 10 bags, spacing the bags equally throughout the lot. The fleeces in these bags were then placed in their proper grade piles, and samples (handfuls) were taken from each fleece, selecting wool from the major body sorts which made up approximately 95% of the fleece by weight. In the case of the graded lots the samples were taken directly as the fleeces were removed from the bags. It has been found that the samples (handfuls) from 50 fleeces make up a sample of convenient size, and thus there were samples for each 50 fleeces in the grade pile. All of the fleeces in each grade pile were sampled. By this method a series of samples were obtained from the sub-lot (10 bags), which in turn were selected at equal intervals throughout the entire lot.

Three different lots were tested (Wyoming Original Bag, Montana Graded ½ Blood, Idaho Graded ½ Blood). The following table shows the structure of these lots, sub-lots, and the samples made up from them:

Lot	Entire Lot No. Bags	Sub-lot No. Bags	Sub-lot No. Samples
Wyoming Original Bag	82	9	6
Montana Graded ½ Blood	126	10	24
Idaho Graded ½ Blood	258	10	12

The number of samples varied because after the first lot was run additional samples were taken in the second lot to test repeated sampling by two operators. In the third lot, duplicate samples were taken by one operator. The samples were taken from the tied fleeces, and then the fleeces were untied and sorted. The mill used only the main body sorts which amounted to 95 to 96% of the total by weight. The off-sorts including tags were not scoured.

In one lot, a corrected yield was calculated for the entire unsorted fleece by obtaining the shrinkage of a sample of tags and correcting for the weight of the tag sort.

The samples were scoured in a semi-commercial type of scouring plant using the common scouring solutions. The yields for both small samples and for the entire lot at the mill were based on a standard moisture content of the scoured wool (12%).

The shrinkage results at the mill were calculated on the basis of 12% moisture, and the yield of top, noils, slubbing and waste which included all of the scoured wool. It was not generally possible to obtain weights of the scoured wool direct, although an attempt was made in one case to obtain

them, and these results were not satisfactory because of the very great variation in the moisture content.

Experimental Results

A comparison of the shrinkage of samples, sub-lots, and entire lots; the shrinkages of samples based on yield of scoured wool at 12% moisture; the shrinkages of mill lots based on yield of top, noil, slubbing and waste at 12% moisture are presented in the following table:

	All Samples		Mill Lots	
	Mean	Std. Dev.	Sub-lot	Entire Lot
Wyoming Original Bag	56.4	1.887	57.0	56.8
Montana Graded ½ Blood	59.7	0.980	60.4	58.5
Idaho Graded ½ Blood	55.6	0.594	54.6	53.4

The differences in shrinkage, using the entire lot as a base, are shown in the following table:

	Sub-lot	All samples
Wyoming Original Bag	+0.2	−0.4
Montana Graded ½ Blood	+1.9	+1.2
Idaho Graded ½ Blood	+1.2	+2.2

The samples showed shrinkages within 2.2% of the entire lot, and it will be noticed that in most cases the shrinkages of the samples were higher than that of the entire lots. The variation of the different samples, as indicated by the standard deviation, was greater in the Wyoming lot with 6 samples; intermediate in the Montana lot with 24 samples, and the smallest in the Idaho lot with 12 samples. These differences in shrinkage between the samples and the entire lot show not only the error of sampling the sub-lot, but also the error in selecting the sub-lot from the entire lot. It is interesting to compare the shrinkages of the samples taken from each sub-lot against the sub-lot. In this case the differences are as follows:

Wyoming Original Bag	−0.6
Montana Graded ½ Blood	−0.7
Idaho Graded ½ Blood	+1.0

The correspondence of the samples and the sub-lots from which the samples were taken is very close and gives encouragement to the program of finding a sampling method which will give representative shrinkages and grades for a larger lot. However, it also points out that, in order to obtain such results, the tags must be removed, which is further confirmed by the results obtained in comparing composite bags and samples taken from them.

There is still the problem of obtaining a sub-lot (approximately 10 bags) which will represent the larger lot. In this study the differences in shrinkage between the sub-lot and the entire lot were all under 2%. In all cases the sub-lots had higher shrinkages than the entire lots. The figure of 2% in difference between samples has been set up as an acceptable difference in so far as practical information to the wool grower is concerned. The three lots tested to date give encouragement to the program of obtaining representative samples from a clip or lot of wool.

In the mill work only the major sorts were processed, and to be applicable to field work in which the entire fleece has been the unit, some allowance would need to be made for tags and the small amount of neck and britch wool that was removed when the wool was sorted at the mill. As mentioned before, the sampling method has been predicated on the basis of grabbing handfuls of wool from the shoulder, side, and back areas of the tied fleece. These areas make up the major part of the fleece. In the mill tests of shrinkage, wool from exactly the same parts of the fleece in the samples was compared with the remainder of the same parts of the fleece when processed. In one lot of wool tested at the mill a sample of tags was taken and the shrinkage of the lot was corrected to allow for the tags. In this case the shrinkage of the lot was increased by 0.6% when the tags were included.

Another basis of comparison of the sub-lots and the entire lots was in the percentage by weight of the different grades in an original lot of 91 bags of Wyoming wool. The results were as follows:

Grade	Percentage of Grades by Weight	
	Entire Lot—82 Bags	Sub-lot—9 bags
Fine	25.85	16.14
½ Blood	66.89	75.58
⅜ Blood	6.77	7.77
Gray	0.49	0.51
Total	100.00	100.00

It will be noted that there was around a 9% difference in the Fine and ½ Blood grades between the entire lot and the sub-lot. The sub-lot, as an indicator of the entire lot, had 9% less of the Fine grade and 9% more of the ½ Blood grade. Thus, it was difficult to pick out representative bags from a lot unless the shearing shed information was known. This was strikingly shown in some grading work carried on at the shearing shed during the 1938 shearing season, in which the first bags shorn from each band contained considerably more medium to coarse wool and less fine wool than the last bag shorn from each band. The information from this clip is shown in the following table:

Grade	Per cent by Weight	
	First Bag in Band	Last Bag in Band
Fine	38.8	67.0
½ Blood	42.2	22.1
⅜ Blood	16.8	10.2
¼ Blood	2.2	0.7
Total	100.0	100.0

Difference in Grading Between Sub-lots and the Entire Clip

During the 1938 shearing season, studies were made of the effect of the grades of fleeces, in the order that they were shorn, on the sampling program. The object of these studies was to find out how representative the different lots of wool were as compared with the total clip in respect to the percentage of each grade. Each fleece in these clips was weighed and graded

as it came to the sacking stand. The results are tabulated and discussed as follows:

Clip No. 1.—5,320 Fleeces, or 97 Bales

Grade	Entire Clip	Per cent of Grades by Weight 10 Bales or Every 10th Bale [1]	5 Bales or Every 20th Bale [1]	Every 10th Fleece
Fine	64.4	65.0	70.2	65.6
½ Blood	25.0	25.1	23.9	22.7
⅜ Blood	9.0	8.2	3.9	10.5
¼ Blood	1.5	1.5	1.6	1.2
Low ¼ Blood	0.1	0.2	0.4	0.0
All Grades	100.0	100.0	100.0	100.0

[1] 10 bales or every 10th bale amount to the same in a 100-bale clip.
5 bales or every 20th bale amount to the same in a 100-bale clip.

It will be noted in the data on Clip No. 1 that every 10th bale or every 10th fleece gave a representative picture of the clip in respect to grading. However, when only five bales were selected at equal intervals they did not give representative results in regard to the grades.

Clip No. 2.—8,376 Fleeces, or 203 Bags

Grade	Entire Clip	Per cent of Grades by Weight 10 Bags	Every 10th Bag	Composite Bag [1]
Fine	82.8	80.8	82.5	72.9
½ Blood	16.2	17.4	16.1	27.1
⅜ Blood	0.9	1.6	1.0	0.0
¼ Blood	0.1	0.2	0.4	0.0
All Grades	100.0	100.0	100.0	100.0

[1] The composite bag was made up by selecting fleeces at equal intervals of time throughout shearing.

In Clip No. 2 every 10th bag gave the best representation of the clip as shown by the per cent by weight of each grade. The composite bag in which fleeces were selected at equal intervals of time throughout shearing of the clip did not give as representative results as the other methods of sampling. Ten bags spaced throughout the clip did not give as good a representation as 20 bags which were selected by taking every 10th bag.

Clip No. 3.—7,649 Fleeces, or 204 Bags

Grade	Entire Clip	Per cent of Grades by Weight 10 Bags	Every 10th Bag	Composite Bag [1]
Fine	80.0	80.0	77.7	80.1
½ Blood	17.8	17.5	19.5	19.9
⅜ Blood	2.0	1.9	2.3	0.0
¼ Blood	0.2	0.6	0.5	0.0
All Grades	100.0	100.0	100.0	100.0

[1] The composite bag was made up by selecting fleeces at equal intervals of time throughout shearing.

In Clip No. 3 both the 10-bag lot and every 10th bag (20-bag lot) gave a good representation of the entire clip in all four grades. The composite bag also gave a good representation in the Fine and ½ Blood grades, but did not contain any ⅜ Blood or ¼ Blood fleeces.

The results in all three of the clips indicate that it does not seem to make much difference in obtaining representative proportions of the different grades, whether a method is used in which every 10th bag is selected or 10 bags spaced equally throughout the clip. This method seems to give just as good results as the selection of every nth fleece throughout the clip to make up 10 bags of wool. The results obtained with these clips of wool indicate that so far as the grading of a clip is concerned the selection of less than 10 bags of wool does not give a representative sample.

Movement of Sheep Into Shearing Pens Affects Sampling

The manner in which sheep move into the shearing pens has a considerable effect on the sampling of a clip where the wool is not graded. It has been a common observation that the coarse-wooled sheep are bolder and tend to be in the lead when the sheep go into the shearing pens, while the fine-wooled sheep tend to "hang back." In the grading work completed on three clips during the 1938 shearing season this tendency was indicated by the grading of the first and last bags sacked from each band as shorn. The first bag of wool from each band of sheep was consistently coarser in grade than the last bag of wool sacked from the same band.

However, there were some factors which tended to complicate this tendency. One of these was the age of the sheep which, irrespective of the grade of the wool, made the older sheep hang back and come in with the last of the band. One of the clips contained fleeces from a number of older "black-face" sheep, and consequently the older sheep with coarser fleeces tended to hang back along with the fine-wooled ones. Where the ages of the sheep were about equal, there seemed to be a strong tendency for the fleeces of the coarser-wooled sheep to be represented in the first bags sacked from a band and the finer wools were found in greater amount in the last bags made up from a band.

The Grease Content of Scoured Wool

The grease content of commercially scoured wool, according to the trade, varies between ½ and ¾ of 1%. Tests of grease content in the wools scoured at the mill and the samples scoured at the Bureau of Agricultural Economics laboratory gave the following results:

	Mill Lots		
	Entire Lot	Sub-lot	B.A.E. Samples
Wyoming Original Bag	0.85	—	0.93
Montana Graded ½ Blood	0.66	0.40	—
Idaho Graded ½ Blood	0.41	0.32	0.61

It will be noted that the grease content of the various scoured wools ranged from 0.32 to 0.93%. There was considerable variation in the grease content of scoured wools, and further work will need to be carried on to determine the normal grease content of scoured wool. The correction of the

shrinkages to a standard grease content of scoured wool (0.5%) made a change in the shrinkages of the mill lots of barely 0.1 of 1%, so the question of grease content in scoured wool is of minor importance in the determination of shrinkages especially when the sampling error was so much larger.

Variation in the Shrinkage Between Four Samples from the Same Fleece

As mentioned in the sampling procedure, some tests were made on the effect of repeated sampling of the same fleeces in the lot of Montana Graded ½ Blood wool, which was the second lot tested at the mill. Two operators took quadruplicate samples from each fleece going over the 50 fleeces twice. All of the odd numbered samples were taken by one operator, and the even numbered samples by the second operator. Thus, samples No. 1 and 3 were taken from one set of 50 fleeces by one operator, and samples No. 2 and 4 were taken from the same 50 fleeces by the second operator. Two different factors were studied in this test, namely, the personal variation in sampling due to different operators and the variation between the first and last samples of four samples taken from the same fleeces. The results obtained are shown in the following table.

Samples by Sets of 4	Shrinkage of Samples Operator No. 1	Operator No. 2	Difference
Set 1	58.3	57.8	0.5
Set 2	60.2	59.3	0.9
Set 3	59.5	60.5	1.0
Set 4	61.3	60.4	0.9
Set 5	60.4	60.0	0.4
Set 6	59.8	58.7	1.1

The difference between operators is not significant, as "t" equals 1.49 which is smaller than the 5% level at 5 degrees of freedom.

An additional study was made of the variation in the shrinkage of the first and last of four samples taken from the same fleeces. In this test, four samples were taken from the same fleece by two different operators. The results obtained with the first (No. 1) and last (No. 4) samples drawn from these fleeces are shown in the following table:

Samples by Sets of 4	Shrinkage of Samples Samples No. 1	Samples No. 4	Difference
Set 1	58.2	57.5	0.7
Set 2	60.1	59.3	0.8
Set 3	59.7	60.3	0.6
Set 4	61.3	60.1	1.2
Set 5	60.4	59.5	0.9
Set 6	60.3	60.0	0.3

The difference between the first and last of four samples as indicated by a statistical analysis of differences is not significant for "t" equals 2.12 which is smaller than the 5% level at 5 degrees of freedom.

Removal of a Sulfur Black Dye from Suspension by Flotation Methods

By B. R. CLANTON,* F. K. CAMERON, and J. E. MAGOFFIN *

A Textile Foundation Publication

Summary

1. It has been confirmed that the flotation of sulfur black suspensions is dependent on the electrokinetic potential of the dye, as measured by the migration velocity.

2. Several new floating agents have been found to be effective alone and in mixtures.

3. Partial coagulation of the dye with electrolytes or positive colloids before addition of floating agents has proved to be effective. The improvement is directly connected with the change in the electrokinetic potential of the dye caused by the coagulating agent.

Introduction

IT has been shown by Clanton[1] that substances in colloidal suspension may be floated successfully by the use of proper collecting and frothing agents. The collecting agent must have a charge or potential opposite to that of the particles to be floated; and it must impart to the particle a water repellent coating or film. Preliminary experiments on several suspensions, including a sulfur black dye made from 2,4 dinitrophenol, already have been reported.[1, 2] The same sulfur black dye was used for the experimental work discussed in this paper.

Experimental

In order to obtain reproducible results more easily, and to approximate more closely usual flotation technique, the apparatus shown in Fig. 1 was constructed. Glass windows were placed in the sides of the cell to permit direct observation of the degree of flotation. Air is admitted into the cell through a hole in the bottom directly under the impeller shaft, and is broken into many small bubbles by the copper grid immediately above the blades of the impeller. Above the impeller the cell is divided into two sections by the perpendicular wooden partition which guards the liquid adjacent to the lip of the cell from agitation and thus permits concentration of solids in the froth.

* Investigators for the Textile Foundation, working under the direction of Dr. Frank K. Cameron at the University of North Carolina.

The volume of froth and rate of froth overflow can be controlled by the amount of air admitted into the cell and by the speed of the impeller. In all experiments to date only the amount of air has been varied, the impeller being run at constant speed. Usually the rate of froth overflow was maintained just fast enough to prevent complete breakdown of the froth and the consequent return of solids to the liquid. The volume of froth and rate of froth formation is, of course, dependent on the reagents used and on the condition of the experiment.

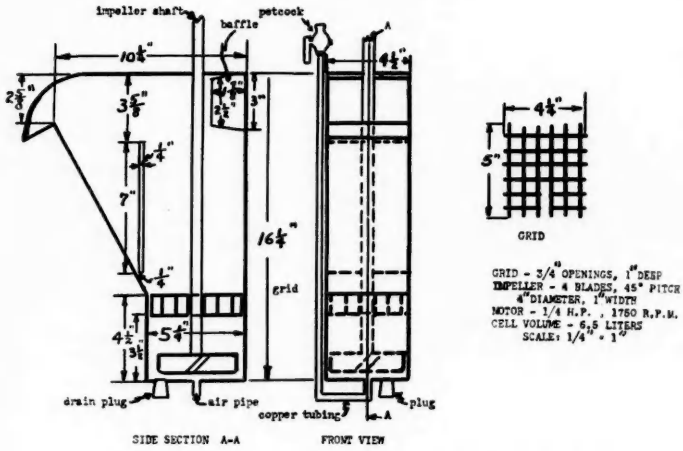

Fig. 1. Inside Dimensions of Laboratory Flotation Machine.

Discussion of Results

Small scale experiments (300 cc. volume of dye suspension) were continued, using several new flotation reagents. The results are listed in Table I, all flotations being complete unless otherwise stated. The data support the conclusions reached from previous experiments, that reduction of the pH and electrokinetic potential of the dye suspension, by the addition of acid, improves the flotation. The effect of added acid is twofold: the hydrogen ions are adsorbed by the dye particles, thus lowering the electrokinetic potential of the particles. This means that less reagent can be adsorbed, since the magnitude of the potential is largely responsible for the adsorption. The lowering of the pH also tends to increase the activity of the positive materials used as flotation reagents, so that a smaller quantity produces the same effect as a larger quantity at a higher pH. If the electrokinetic potential of the particle is reduced sufficiently by the adsorbed hydrogen ions, coagulation of the dye suspension occurs. The lower charge requires less flotation agent for complete neutralization, and the coagulation tends to produce a heavier and less dilute froth. The second effect is of particular

importance for waste control, since the success of a waste removal process lies in the separation of the dispersed material from the dispersion medium.

Of the reagents listed in Table I, "D. P. 243," a quaternary ammonium salt made by E. I. du Pont de Nemours and Co., gives the best results. Monoamylamine causes as complete a removal, but the froth is far more dilute. "Zephiran," another quaternary ammonium salt, made by the Alba Pharmaceutical Company, is a good precipitation agent for sulfur black, but lacks the ability to collect the particles in a stable froth. Since monoamylamine causes excessive frothing, it was hoped that a mixture of it and Zephiran might prove more efficacious than either alone.

TABLE I

pH	u in μ/sec./volt/cm.	Floating Agent	Volume	Remarks
10.40	6.71	Monoamyl-amine	0.2 cc.	Excessive froth
7.98	5.18	"	0.1 cc.	Froth more concentrated
4.08	—	"	0.1 cc.	Froth more concentrated
10.40	6.71	Diamylamine	0.4 cc.	Excessive froth not quite complete
7.45	4.80	"	0.3 cc.	Better froth
10.40	6.71	Triamylamine + 1N HCl	0.3 cc. 5.0 cc.	Not quite complete
7.60	5.00	"	0.2 cc. 0.2 cc.	" " "
10.40	6.71	"D. P. 243" 1% solution	3.0 cc.	
7.32	4.75	"	2.1 cc.	
10.40	6.71	"Hyamine"	0.45 cc.	Incomplete
10.40	6.71	"Zephiran"	16.0 cc.	Incomplete and poor froth Dye is pp't.

In Table II are given the data for various mixtures of the amine and "Zephiran," and for the amine and "Hyamine," the volume of the dye suspension being 300 cc. Again all flotations are complete unless otherwise stated. It was found that the mixture of monoamylamine and "Zephiran" is better than either agent used alone. In two experiments there was an actual reduction in the amount of reagent needed for complete flotation, and in all four there was a decided improvement in the quality of the froth, the 75% monoamylamine-25% Zephiran mixture producing the most concentrated froth. The mixtures of monoamylamine with "Hyamine" were not very effective.

The data in Table I suggested that better flotation is obtained when the dye is subjected to coagulation before addition of the floating agent. Best results ought to be obtained in the case of sulfur black by using high valency cations as coagulation agents, since the dye is charged negatively

TABLE II

pH	u in μ/sec./volt/cm.	Floating Agent		Vol.	Remarks
10.40	6.71	Monoamylamine 90%	"Zephiran" 10%	0.21 cc.	Excessive froth
10.40	6.71	75	25	0.4 cc.	Concentrated froth
10.40	6.71	50	50	0.35 cc.	Fair froth
10.40	6.71	25	75	0.9 cc.	Poor froth
10.40	6.71	Monoamylamine 75	"Hyamine" 25	0.25 cc.	Poor froth
10.40	6.71	50	50	0.4 cc.	Poor froth
10.40	6.71	25	75	0.45 cc.	Poor froth and incomplete Flotation

TABLE III

Salt added gm./liter	pH	u in μ/sec./volt/cm.	Floating Agent	Vol.	Remarks
$Al_2(SO_4)_3 \cdot 18\ H_2O$					
0	10.40	6.71	Monoamylamine	1.2 cc.	Excessive froth
0.044	7.50	4.28	"	1.2 cc.	Froth becomes more concentrated
0.055	7.34	4.00	"	1.2 cc.	"
0.065	6.30	1.50	"	3.0 cc.	"
0.088	5.80	0.34	"	1.2 cc.	"
0	10.40	6.71	"D. P. 243"	18 cc.	Very good
0.119	—	—	"	17 cc.	Better froth
$Fe_2(SO_4)_3 \cdot 6.3\ H_2O$					
0.16	4.15	1.96	Monoamylamine	1.2 cc.	Good froth
0.16	4.15	1.96	Monoamylamine + "Zephiran" 75–25	4 cc.	Poor froth
0.16	4.15	1.96	"D. P. 243"	14 cc.	Concentrated froth, rapid flotation
$Cr_2(SO_4)_3$					
0.0876	6.0	—	"D. P. 243"	15 cc.	Excellent froth
0.15	—	—	"	50 cc.	No flotation
0.11	—	—	Monoamylamine + "Zephiran" 75–25	4.5 cc.	Incomplete flotation

in suspension. The sulfates of iron, aluminum, and chromium were selected because of availability.

The dye suspension was prepared in the usual manner, the salt being added to the suspension during violent agitation, which continued for ten minutes before any floating agent was added. To facilitate better mixing, the salts were added in freshly prepared solutions.

In Table III are listed the data for flotations of electrolyte conditioned suspensions, all run in the 6.5 liter cell. In all cases the flotation was complete unless otherwise stated. Increasing the amount of $Al_2(SO_4)_3 \cdot 18H_2O$ in the suspension decreases both the migration velocity and the pH, and although there is no noticeable change in the amount of floating agent required for complete flotation, there is a marked improvement in the quality of the froth with these changes. The froth becomes increasingly concentrated and the flotation more rapid with increasing concentration of aluminum. With "D. P. 243" as floating agent the dye from 6.5 liters can be concentrated in about 100 cc., when the aluminum salt is used.

Iron salts are not as good coagulating agents for this particular dye, the flocs being much smaller than with aluminum and chromium salts. Flotations are but slightly better than when no salt is used.

Chromium sulfate proves very effective when used with "D. P. 243" as floating agent. There is a reduction in the amount of "D. P. 243" required for complete flotation as well as a decided improvement in the character of the froth. However, it is apparent that the addition of too much coagulating agent is detrimental to the flotation process. Complete or nearly complete coagulation with chromium sulfate prevents flotation with even 50 cc. of "D. P. 243." This failure is undoubtedly due to the fact that the adsorbing power of the dye particles for the floating agent

TABLE IV

Positive Sol.	Sols. Vol.	Added pH	u in μ/sec./volt/cm.	Floating Agent	Volume	Remarks
S.B+Fe_2O_3						
90–10	300 cc.	9.55	5.44	Monoamylamine	0.1 cc.	Heavy froth Rapid flotation
90–10	300 cc.	9.55	5.44	Aniline + 1N HCl	8.0 cc. 0.7 cc.	Dye pp't. but incomplete flotation
90–10	300 cc.	9.55	5.44	Naphthylamine 3% + 1N HCl	1.6 cc. 3.0 cc.	Complete flotation but slow
90–10	6000 cc.	9.55	5.44	Monoamylamine	1.0 cc.	Excessive froth
S.B+Cr_2O_3						
90–10	300 cc.	9.88	3.10	Monoamylamine	0.2 cc.	Excessive and dilute froth
70–30	300 cc.	9.13	3.18	"	0.17 cc.	Better froth
60–40	300 cc.	8.73	3.13	"	0.1 cc.	Good froth
70–30	6000 cc.	8.73	3.18	"	0.7 cc.	Excessive froth

is proportional to the negative potential of the particles. When this potential is completely or nearly completely removed, the particle can no longer adsorb the reagent, and thus does not acquire a water repellent coating.

The effect of positive colloids on flotation of sulfur black was also studied, and the results assembled in Table IV. The particular mixtures studied were stable ones, the concentration of the positive sol being kept below the coagulating point. Comparison of these flotations with those of sulfur black alone and with those of sulfur black conditioned with added electrolytes reveals that the addition of a positive sol causes only a slight improvement over straight dye flotations, but is much less effective than addition of electrolytes containing high valency cations.

References

1. B. R. Clanton. *Text. Rsch.*, *8*, No. 8, 220 (1938).
2. J. E. Magoffin and B. R. Clanton. *Ibid.*, *8*, No. 10, 357 (1938).

Abstracts

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Address, C. H. Clark, Sec., 65 Franklin St., Boston, Mass.

I. Fibres (Synthetic and Natural) and Fibre Analysis

Lattice Structure of Native Cellulose: Investigation of the. H. Kiessig. *Z. physikal. Chem.*, May 1939, V. 43, No. 2, P. 79–102; abs. in *Eastman Kodak Abs. Bull.*, Oct. 1939, P. 536.

From a critical examination of the X-ray diffraction evidence, it is concluded that the correct lattice model has not yet been established. Certain odd interferences do not agree with the Meyer and Mark model. (S)

Ontogenesis and Chemical Structure of the Vegetable (Cotton) Cell Wall. Hess, Wergin, Kiessig, Engel and Philippoff. *Naturwis.*, 1939, V. 27, P. 622–8; abs. in *C. A.*, 1940, V. 34, Col. 259.

A review of development and structure of cotton fibres, differences between the phases of length growth and thickness growth, the change of cellulose from unordered form to regular mol. arrangement as found by X-ray and the presence of waxy material in the young fibre. The qualities of the primary and secondary wall materials are compared in some detail. Whereas in the primary stage (length growth) waxes, phosphatides, protein, etc., are formed in the walls, the second stage is primarily one of formation of cellulose lattice. The primary cellulose, present in small amts., is not in lattice form owing to the presence of the admixts.; removal of the latter spontaneously causes change to lattice form (X-ray, double refraction, swelling and viscosity evidence). (C)

PROTEINS OF SILK (FROM THE DEGUMMING TO THE PRODUCTS OF HYDROLYSIS). M. Jarach. *Boll. ufficiale staz. sper. seta*, 1937, V. 7, P. 71–4; *Chem. Zentr.*, 1938, V. i, P. 2281; abs. in *C. A.*, 1939, V. 33, Col. 9000.

A review is given of the most important amino acids formed by the hydrolysis of fibroin and sericin. (C)

PROTEIN RAYON: PRODUCTION IN JAPAN. I. Sakurada. *J. Soc. Chem. Ind. Japan*, 1939, V. 42, P. 191–2B; abs. in *J. T. I.*, Nov. 1939, P. A689.

A brief review is given of the possibilities of using soy bean and fish proteins as sources of fibre of the "Lanital" type. The Showa-Sangyo firm is marketing a soy bean protein fibre under the name "Silkool." Tensile properties are recorded for five samples, four about 5.5 den., breaking loads of about 0.5 gm. per den. (dry), and extensions of 3–16%, and one of 10 den. with 61.5% extension. Tensile data are recorded for other soy bean and fish protein fibres, in comparison with Lanital, Tiolan, wool and viscose staple fibre. An amino-acid analysis is tabulated for wool keratin, casein and soy bean protein ("glycinin"). (C)

RAMIE AND NITRORAMIE: COMPARISON OF STRENGTHS OF. I–II. I. Sakurada and S. Kawada. *J. Soc. Chem. Ind. Japan*, Suppl. binding, 1939, V. 42, P. 225–6; abs. in *C. A.*, 1939, V. 33, Col. 8999.

Nitroramie was compared with partially treated ramie, rather than with the crude ramie, to det. whether the differences are due to esterification or to soln. of the raw material. Five kinds of ramie were studied. On the basis of the results it is concluded that esterification has no influence on the gross strength of individual fibres, and only causes an increase in thickness, the elongation remaining practically the same. Arguments are advanced against the theories of H. Mark, K. H. Meyer and J. H. de Boer relating to the strengths and structures of artificial fibres. (C)

RETTING OF FIBRE FLAX. B. B. Robinson. *Linen Trade Circ.*, 1939, V. 5, No. 9, P. 8, 10, 15; abs. in *J. T. I.*, Nov. 1939, P. A688.

Discusses results obtained from recent research work on the retting of flax in the U. S. A. In this article the warm water tank retting method as used in Oregon is compared with the dew retting and chemical retting of flax straw. Statistics for retting in tanks are given. (C)

SERICIN. V. CHANGES OF SILK PROTEINS BY HEATING WITH WATER. T. Ito and K. Komori. *J. Agr. Chem. Soc. Japan*, 1939, V. 15, P. 300–4; *Bull. Agr. Chem. Soc. Japan*, 1939, V. 15, P. 46–8 (in German); abs. in *C. A.*, 1939, V. 33, Col. 4793; 1934, V. 34, Col. 270. (C)

SYNTHETIC FIBRES: PUTTING THE SPOTLIGHT ON THE NEW. *Text. Wld.*, Sept. 1939, P. 74–80.

The properties and uses of the new fibres are described. (C)

II. YARNS AND FABRICS

SHUTTLE PROJECTION: MATHEMATICAL THEORY OF. J. J. Vincent. *J. T. I.*, July 1939, P. T103–26.

The position, speed, and acceleration of the shuttle are calculated in terms of loom parameters, the elasticity of the picking mechanism being

taken into account but its mass neglected. Various types of nominal movement are considered—those in actual use, those suggested in text books, and others that seem to be an improvement on current practice. The "catapult action" observed at the British Cotton Industry Research Association and elsewhere is quantitatively explained and a general theorem giving an upper limit to its effect is deduced. Other results of value to the overlooker and designer are also given. The effect of the mass of the picking mechanism is briefly considered, and the theory of a simple analogous system is given in an appendix. (C)

SILKY, SMOOTH FINISHES: TROUBLE WITH. *Wool Record & Text. Wld.*, Sept. 21, 1939, P. 604, 606.

A practical treatise on the finishing of wool covert repps. (C)

SIZING: DON'T OVERLOOK THAT WATER IS AN INGREDIENT IN. S. G. Davenport. *Rayon Warp Sizing Specialist*, Sept. 1939.

Impure water is one of the frequent causes of sizing trouble and the author notes a number of the sizing troubles resulting therefrom. (C)

SOY BEAN TEXTILE FIBRES: FORD RESEARCH IMPRESSIVE. Stephen S. Marks. *Daily News Record*, Aug. 22, 1939, P. 13. (C)

SPUN RAYON WARP SLASHING TECHNIC. W. L. Bentley. *Textile Age*, Oct. 1939, P. 36, 38, 40–1.

The author states that neither the materials nor methods used in the warp sizing of cotton or filament rayon are satisfactory for spun rayon warps, and discusses those that he has found satisfactory. (C)

STAPLE FIBRE YARNS: SIZING. *Spinner u. Weber*, 1939, V. 57, No. 24, P. 5–6; abs. in *J. T. I.*, Aug. 1939, P. A514.

The use of staple fibre in mixtures with other fibres, especially with wool, is discussed and various general practical hints are given on the processing of staple fibre. A suitable size for staple fibre warps contains 5 kg. potato flour, 1 kg. fat and 1 kg. glycerine and a suitable additional product per 100 l. A sizing machine with an air drying system should be used and the temperature should not rise above 70° C. in the drying chamber nor be higher than 65° C. in the sizing bath. The warp yarns should not be immersed in the sizing bath but should pass directly from the warp beam to the sizing and squeeze rollers, since staple fibre yarns require mainly a surface sizing and care must be taken to avoid stretching in the wet state. Automatic tension-regulating means should be provided. (C)

STAPLE QUALITY VS. YARN QUALITY. E. H. Helliwell. *Textile Age*, Aug. 1939, P. 39–40.

The author holds that old warnings against mixing bales of cotton of varying length and character do not hold good with modern blending equipment and high-draft spinning.

STRENGTH OF KNITTED FABRICS UNDER INCREASING YARN TWIST. W. Davis. *Text. Mfr.*, Aug. 1939, P. 342, 345. (C)

TECA: ITS PROPERTIES AND UTILIZATION. Harold DeWitt Smith. *J. T. I.*, July 1939, P. P153–61.

Teca is the name of a family of cellulose acetate rayon staple fibres manufactured by the Tennessee Eastman Corp. Teca fibres are characterized by an inherent crimp which, in combination with the resilience of the fibre substance, imparts a distinctive lofty character to yarns and fabrics in which they are present. The properties and methods of processing Teca into yarns and fabrics are discussed. The types of fabrics in which Teca is being effectively used are described. (C)

"TENASCO" FOR RAYON TIRE CORD, ETC. L. Rose. *J. T. I.*, July 1939, P. P202–12; *Text. Mfr.*, June 1939, P. 248–9; *Text. Recdr.*, June 6, 1939, P. 27–8.

"Tenasco" is the registered name given by Courtaulds, Ltd., to a new class of high tenacity yarns made by a modification of the ordinary viscose spinning process and which are finding increasing uses for industrial purposes, thus bringing rayon into a field of enterprise which hitherto it was thought impossible for it to enter. (C)

TENSILE STRENGTH OF YARNS. P. Larose. *Canadian Text. J.*, Nov. 10, 1939, P. 31–3.

The first of two instalments describing the basic factors of fibre quality, twist, doubling, and fibre distribution which influence and determine the strength of cotton yarns. (C)

TWIST: AMOUNT OF. *Am. Wool & Cotton Rptr.*, May 11, 1939, P. 59–60.

Indicates that twist now commonly employed for spun rayon warps is higher than that which gives the strongest yarns. (C)

TWIST AND LUBRICATION OF SILK HOSIERY YARNS. W. Davis. *Rayon Text. Mo.*, Aug. 1939, P. 47–9. (C)

WASHING-OFF PROCESS IN SCOURING (of Wool Cloth). J. Colin Schofield. *Wool Record & Text. Wld.*, Sept. 14, 1939, P. 556–7, 563. (C)

WOOL CARBONIZING. Robert R. Sleeper. *Am. Wool & Cotton Rptr.*, Sept. 28, 1939, P. 11–12, 21–2.

Discussion on the sulphuric acid, aluminum chloride and hydrochloric acid processes, with particular reference to their effect on the dyeing properties of the carbonized product. (C)

(WOOL) CLOTH SCOURING PLANT: MANAGEMENT OF A. *Wool Record & Text. Wld.*, Sept. 7, 1939, P. 506–7.

The practical difficulties that are associated with the process of cloth scouring: production, time, nature of the pieces treated, and steam, water and assistants used in the scour. The supply of steam from the point of view of both quantity and quality is mainly responsible for whether or no scouring is successful. It is, in fact, just as important as the water supply. (C)

WOOL DRYING PROBLEMS. *Wool Record & Text. Wld.*, Sept. 7, 1939, P. 503–4.

Of chief value as an exposition of the crude rule-of-thumb methods by which practical men try to dry efficiently and safely. (C)

YARN COUNT VARIATIONS: REDUCTION. G. Schmalfuss. *Klepzig's Textil Z.*, 1939, V. 42, P. 317–9; abs. in *J. T. I.*, July 1939, P. A451.

It is pointed out that variations in yarn counts are more frequent with high-draft systems than with ordinary drafts and the reason for this fact is explained. The case of a 40s yarn showing variations over a range amounting to 36.75% of the theoretical counts is studied. Examination of slivers and roving at different stages showed the irregularity to be present in the roving to have originated mainly in the drawing process. By reducing the speed of the front roller, using three passages of drawing, 8-fold instead of 6-fold doubling, and producing a finer sliver on the drawframe it was found possible to reduce the range of variation in the final yarn to 13.75%. (C)

III. CHEMICAL AND OTHER PROCESSING (NOT OTHERWISE CLASSIFIED)

ACID WOOL DYES: IMPARTING AFFINITY FOR—TO REGENERATED CELLULOSE FIBRES BY INCORPORATING SYNTHETIC RESINS IN THE SPINNING SOLUTION. T. Crebert. *Zellwolle*, 1939, V. 5, P. 148–56, 182–7, 227–31; abs. in *J. Soc. Dyers Col.*, Oct. 1939, P. 517; *J. T. I.*, Nov. 1939, P. A689. (C)

ANIMALIZED CELLULOSE FIBRES: AFFINITY FOR ACID WOOL DYES. P. Eckert, E. Herr, and G. Fischer. *Klepzig's Textil-Z.*, 1938, V. 41, P. 624–7, 634–7, 646–7; 1939, V. 42, P. 164–8; abs. in *J. T. I.*, May 1939, P. A323.

An account is given of investigations of the influence of the pH, volume, and concentration of the dye bath, time of dyeing, etc., on the adsorption of acid wool dyes by animalized viscose fibre. Triphenylmethane and azo dyes were used in the experiments. The results are given in tables and graphs and are discussed. They show that the affinity of animalized fibres for acid wool dyes, like that of wool, increases with decreasing pH. (C)

ARIDYE PROCESS OF PRINTING. Norman S. Cassell. *Am. Dye. Rptr.*, June 26, 1939, P. 341–3.

Paper presented at meeting, R. I. Section, A. A. T. C. C., Jan. 27, 1939. (C)

AZOIC DYES. J. A. Wallwork. *J. Soc. Dyers Col.*, Oct. 1939, P. 477–80.

Methods for the application to wool and cellulose acetate rayon are given. (C)

CELLULOSE ACETATE RAYON: DYEING. P. Müller. Imperial Chemical Industries, Ltd. *Text. Wkly.*, 1939, V. 23, P. 739–40; abs. in *J. T. I.*, July 1939, P. A467.

A discussion of methods of dyeing cellulose acetate rayon materials and materials containing this type of rayon in combination with cotton or regenerated cellulose rayon, details of the procedures and necessary precautions, various difficulties and the methods by which they may be overcome, and methods of correcting unsatisfactory dyeings and stripping dyes from cellulose acetate rayon. (C)

CELLULOSE: DEPOLYMERIZATION. H. J. Henk. *Zellwolle*, 1939, V. 5, P. 43–5; abs. in *J. T. I.*, May 1939, P. A339.

A general account is given of the actions of acids, alkalis, salts and enzymes on cellulose, and the decomposition resulting from oxidation of cellulose in bleaching processes, by the action of peroxides formed from dyes, textile assistants, linseed oil, etc., and on exposure to ultra-violet light. The depolymerization of cellulose occurring under the influence of such agents and on strong heating is discussed and suggestions for its avoidance in finishing processes are made. The greater resistance of the higher-polymer celluloses to the various reagents and treatments is pointed out. (C)

COTTON-STAPLE FIBRE MIXTURES: ACTION OF ALKALIS ON. L. Kollmann. *Monatsch. Seide u. Kunstseide*, 1939, V. 44, P. 142–8; abs. in *J. T. I.*, July 1939, P. A466. (C)

DAMAGE TO WOOL BY BACTERIA: PREVENTION OF. R. Hünlich. *Textil Betrieb*, July 1938, P. 41 (through *C. A.*, 1939, V. 33, Col. 1949); abs. in *J. T. I.*, May 1939, P. A325.

Wool can be rendered more resistant to bacterial attack by treatment with chromium oxy salts, *e.g.*, basic chromium magnesium sulphate, if necessary, in the presence of bisulphite, chromic acid, potassium bichromate, zinc salts, etc., at 50° C. (C)

DETERGENT PROPERTIES OF ETHER SULPHONATES. F. J. van Antwerpen. *Ind. Eng. Chem.*, 1939, V. 31, P. 64–6.

The physical properties of a new commercial sulphonated aliphatic ether are described. Graphs illustrating the superiority of this agent in detergent efficiency in comparison with soap, sulphated alcohols and sulphonated esters, are appended. Owing to its remarkable stability, the product is capable of wider industrial application and several new uses are suggested. (C)

DULL SHADES: PREVENTION OF—DUE TO THE PRESENCE OF IRON IN THE DYEBATH, WHEN DYEING WITH CHROME DYES. I. IRON IN THE WATER SUPPLY. C. L. Bird and E. Molloy. *J. Soc. Dyers Col.*, Nov. 1939, P. 560–4.

The following compounds have been shown to possess in varying degree the property of sequestering small amounts of dissolved iron present in the dyebath, sufficiently to prevent the formation of iron lakes when dyeing with chrome dyes: Calgon T, acid sodium pyrophosphate, Trilon B (IG), tetrasodium pyrophosphate, and sodium tripolyphosphate. The effect of varying conditions on the efficacy of these compounds has been studied. It has been confirmed that, in the metachrome and chrome in the bath processes, the presence of potassium bichromate or chromate throughout the dyeing process affords good protection against iron. The protection is less effective when the chromium is present on the fibre, *i.e.*, when dyeing on a chrome mordant. (C)

DYEING OF NYLON. G. S. J. White. *J. Soc. Dyers Col.*, Aug. 1939, P. 409–12.

Acetate rayon dyes present the readiest method of obtaining level shades of reasonable fastness to washing and light on nylon, especially for hose and

lingerie. Where a higher degree of washing fastness is required, *e.g.*, sewing thread, the acid wool dyes will be found valuable. Dyeings of extreme light fastness may be met with vat and Soledon dyes. (C)

IV. Research Methods and Apparatus

Stereophotomicrographic Methods. B. Wright. *Phot. J.*, September 1938, V. 78, P. 597–600; abs. in *Eastman Kodak Abs. Bull.*, April 1939, P. 212.

The author claims that the present practice of stereophotomicrography by both the tilt and side-shift methods is incorrect, since it is based on Müller's fallacy that stereoscopy is the result of a triangulation of the eyes and that distance is measured by the convergence. He recommends a total shift on the screen of ½ mm. to each side, or to give a convergence of ¼ degree.

Testing Staple Fibre: New Methods of Testing. Otto Eisenhut. *Mell. Textilber.* (German edition), 1939, No. 9, P. 625.

It is pointed out, in particular, that the strength of itself by no means represents a decisive factor for the weaving qualities of the material, but rather the elastic elongation of the fibre, upon which the elastic behavior of the fabric to a large extent depends. The article reproduces a number of elongation diagrams produced by means of a newly designed apparatus that is shortly to be placed on the market. It is further pointed out that not only the determination of the mean degree of polymerization, but also the determination of the fractionated degree of polymerization, are necessary when investigating the degree of polymerization of staple fibre, whereby the distribution of the molecular lengths in the fibre is brought out. It has been found that the solubility of staple fibre in alkali is reduced when the lower fractions of the degree of polymerization are discarded. Owing to the removal of the short chains of cellulose, the viscose types of staple fibre made by the Lanusa process (stretch spinning process) offer good resistance to alkali. (C)

Textile Fibres: Staple Diagrams and Mean Fibre Length. H. Köb. *Klepzig's Textil-Z.*, 1939, V. 42, P. 342–50; abs. in *J. T. I.*, Aug. 1939, P. A541.

The preparation and use of staple diagrams, the significance and determination of mean fibre length (or frequency-mean staple) and weight-mean staple, and the influence of fibre fineness on mean length determinations are studied in relation to the recommendations published in certain sections of Normblatt DIN DVM 3801. The recommendations are criticized, especially in regard to the expressed preference for the use of the fibre-weight curve rather than the fibre-number curve. Passages from the proposed standard are quoted and errors in them and confusion in the use of terms are pointed out. A suggested improved version is given. (C)

Textile Finishing Treatments: Technical Evaluation of. Lelia J. Winn and Edward R. Schwarz. *Text. Rsch.*, Nov. 1939, P. 5–16; *Am. Dyc. Rptr.*, Nov. 27, 1939, P. 688–94.

A series of ten fabrics, ranging from organdie through tracing cloth to a thick, stiff-finished cotton material, and varying in weight from approxi-

mately 5 milligrams per square centimeter (1.46 ounces per square yard) to approximately 45 milligrams per square centimeter (13.25 ounces per square yard), have been compared for bending length, rigidity, bending modulus, bending torque, chord length and radius of curvature by means of the drape tester, the hanging loop, Gurley stiffness tester and the Schiefer flexometer. The tests were all conducted in an atmosphere closely approximating standard conditions after adequate conditioning of the samples in this same atmosphere. (C)

TWIST TESTER. G. Krauter. *Klepzig's Textil-Z.*, 1939, V. 42, P. 350–1; abs. in *J. T. I.*, Aug. 1939, P. A543.

Photographs are given of a Zweigle twist tester and the method of use is outlined. It is claimed that with this device continuous tests can be made along the yarn leaving practically no gaps between successive sections tested, that pushing back of the twist when introducing yarn into the clamps, the influence of handling, and arbitrariness in the selection of yarn sections for testing are eliminated, and a uniform loading is ensured. (C)

VARIABLE-TITRE RAYONS: TESTING. P. A. Koch. *Klepzig's Textil-Z.*, 1939, V. 42, P. 143–51; abs. in *J. T. I.*, May 1939, P. A339.

The production of rayon threads showing variations in thickness is discussed and the desirability of an irregular distribution of the thick and thin places is pointed out. The variations in thickness can be followed by means of the Gleifometer, and the Frenzel-Hahn continuous tester is also useful in the study of such yarns. Records obtained with these instruments in tests on samples of various yarns are reproduced and discussed. (C)

WATER REPELLENCY AND TEMPERATURE. H. W. Stiegler and J. M. Hood. *Am. Dye. Rptr.*, May 29, 1939, P. 285–7.

Describes and illustrates apparatus and method for spray and hydrostatic testing, emphasizing the need of accurate control of water temperature. (C)

WETTING AGENTS: EVALUATION OF. Joseph W. Creely and George Le Compte. *Am. Dye. Rptr.*, Aug. 7, 1939, P. 419–20.

In conclusion the authors state that each problem in wetting out tends to be unique and cannot in general be solved by one standard test applicable to all wetting problems. Tests on wetting agents to be used in a given process must be planned with the application in mind. The type of cloth or fibre, the pH of the solution, the concentration of the wetting agent, the temperature, the methods of manipulation, all should correspond as closely as possible with actual practice. (C)

WETTING AGENTS: CONSIDERATION OF THE A. A. T. C. C. OFFICIAL AND THE CANVAS DISC METHODS FOR EVALUATION OF. (Revised.) Carl Z. Draves. *Am. Dye. Rptr.*, Aug. 7, 1939, P. 421–4.

Paper presented at annual meeting, A. A. T. C. C., Atlanta, Ga., Dec. 2, 1938, and recently revised. (C)

WETTING AGENTS: EVALUATION OF. The official A. A. T. C. C. Method. Carl Z. Draves. *Am. Dye. Rptr.*, Aug. 7, 1939, P. 425–8. (C)

V. Pure Science, Economics and Misc.

Ancient Fabrics and Paper: Degree of Polymerization of Cellulose. H. Staudinger and F. Reinecke. *Mell. Textilber.*, 1939, V. 20, P. 109–10.

Tables are given showing the results of strength tests on mummy cloths, other ancient cotton and linen fabrics, papyrus and paper, and the average degrees of polymerization of the cellulose of these materials before and after dissolving in and precipitating from cuprammonium solution. The average degrees of polymerization were above 200. At degrees of polymerization below 200 cellulose disintegrates, forming a fine powder, and loses its capacity for fibre formation. (C)

Artificial Daylight for Color Grading of Agricultural Products. D. Nickerson. *J. Opt. Soc. Am.*, January 1939, V. 29, P. 1–9; abs. in *Eastman Kodak Abs. Bull.*, August 1939, P. 426.

The development of methods and types of illumination is described. For the grading of cotton, it was found necessary to provide a nearly uniform diffuse illumination over the working area, of a color to match a slightly overcast north sky and an intensity on the horizontal working plane of from 60 foot-candles to 80 foot-candles. (S)

Cellulose Fibres: Absorption of Alkali, and Swelling. Gi-iti Saito. *Kolloid Beihefte*, 1939, V. 49, P. 365–454; abs. in *J. T. I.*, Aug. 1939, P. A539.

The author presents a long series of measurements of the swelling (cross-section) and length changes of and absorption by cotton, ramie and rayon fibres in various solutions of Na, K and Li hydroxides, at various temperatures. The experimental technique is described in detail but follows with minor modifications well-known methods. The results are given in 52 tables and 43 sets of graphs. (C)

Cellulose: Reaction with Alkali. T. Lieser, L. Henrich and F. Fichtner. *Liebigs Ann. Chem.*, 1939, No. 538, P. 99–109; abs. in *J. T. I.*, July 1939, P. A498.

The "mercerization" reaction has been studied by steeping bleached lintners or card sliver in the alkali, washing the mass with an alcohol in an atmosphere of nitrogen until the filtrate was substantially free from alkali, drying the residue over caustic potash and phosphorus pentoxide in a high vacuum, and determining the alkali content. The results are discussed on the basis that the reaction is one between the alkali hydroxide and hydroxyl groups in the micelle surface. (C)

Chain Structure of Linear Polyesters: Further Investigation of the. G. C. S. Fuller and C. J. Frosch. *J. Phys. Chem.*, March 1939, V. 43, P. 323–34; abs. in *Eastman Kodak Abs. Bull.*, July 1939, P. 384.

X-ray diffraction investigations on orientated fibres of the ethylene polyesters of succinic, adipic, suberic, azelaic, and sebacic esters, and on the polyester of ω-hydroxydecanoic acid are described. The result, *i.e.*, the fibre period is less than the theoretical, is ascribed to a shortening in the ethylene glycol portion of the chain. In the case of the ethylene succinate polymer, two different crystallographic forms may be identified. (S)

STARCHES, MODIFIED: PART II. DEGRADATION OF STARCHES BY HYPOCHLORITE SOLUTIONS. G. B. Jambuserwala and K. R. Kanitkar. *J. T. I.*, May 1939, P. T85–98.

In a previous study by the same authors of starches modified by means of varied quantities of potassium dichromate and sulphuric acid, it was found that the majority of starches were degraded to such an extent that they could be of very little use for textile processes. In continuation of the work starches have been treated with various concentrations of neutral hypochlorite solutions. The degrading action in these circumstances is due to the oxidizing agent alone since no acid is present. When the reducing values of starches modified by the two oxidizing agents, having the same oxygen content, are compared, it is surprising to find that modification by means of potassium dichromate affects the reducing value about three times as much as modification by means of hypochlorite solution. Thus a reduced degree of modification is effected in the present series of modified starches with a view to their applicability to textile operations.

In the summary of results it is stated that the modified starches produced in the investigation have a greater penetration; in other words these have smaller deposition on the surface and as such could result in decreased wear and tear of the sized fibre. The lower viscosities of the modified forms render possible the use of lager quantities of starch in the mix without increasing the viscosity beyond working limits. Also the sized fibre acquires a softer feel and it is more adaptable to textile operations when modified forms of starch are substituted for the raw variety. (C)

SURFACE-ACTIVE AGENTS: DETERMINATION OF ACTIVE INGREDIENTS AND FATTY MATTER IN. Ralph Hart. *J. Ind. Eng. Chem., Anal. Ed.*, Jan. 15, 1939; *Am. Dye. Rptr.*, May 29, 1939, P. 297–9; *Rayon Text. Mo.*, June 1939, P. 69–70.

TEXTILE ENGINEER'S PLACE TOMORROW. Fredk. M. Feiker. *Lowell Text. Inst. Bulletin*, Aug. 1939.

Commencement address at Lowell (Mass.) Textile Institute, June 6, 1939. (C)

TEXTILE RESEARCH: PROGRESS IN—FROM THE CONSUMER POINT OF VIEW. Pauline Beery Mack. *Am. Dye. Rptr.*, Nov. 27, 1939, P. 696–9.

Restricted to research studies on textiles which have been planned and carried out distinctly from the consumer point of view. (C)

ULTRA-VIOLET LAMPS: USE IN THE TEXTILE INDUSTRY. F. Ohl. *Kunstseide*, 1939, V. 21, P. 162–4; abs. in *J. T. I.*, July 1939, P. A484.

Apparatus for the production of ultra-violet light is briefly described and luminescence, fluorescence and phosphorescence are discussed. The use of ultra-violet light, filtered by the Wood method or through a black glass filter, for the detection of oils, finishing agents, etc., and the identification of dyes on textile materials is discussed and a marking ink which is visible in ultra-violet light but not in ordinary light is mentioned. (C)

UNIFORM HUMIDITY AND COOLING: MODERN AIR CONDITIONING FEATURES. Robert H. Brown. *Cotton*, Aug. 1939, P. 57–9. (C)